

Surface Characterization Session

- Surface Characterization Talk – Christine Trepanier, NDC ~ 40 min
- Objective 1: Homework and Discussion – Matthew Di Prima, FDA ~ 40 min
- Objective 2: Homework and Discussion – Dave Saylor, FDA ~ 40 min

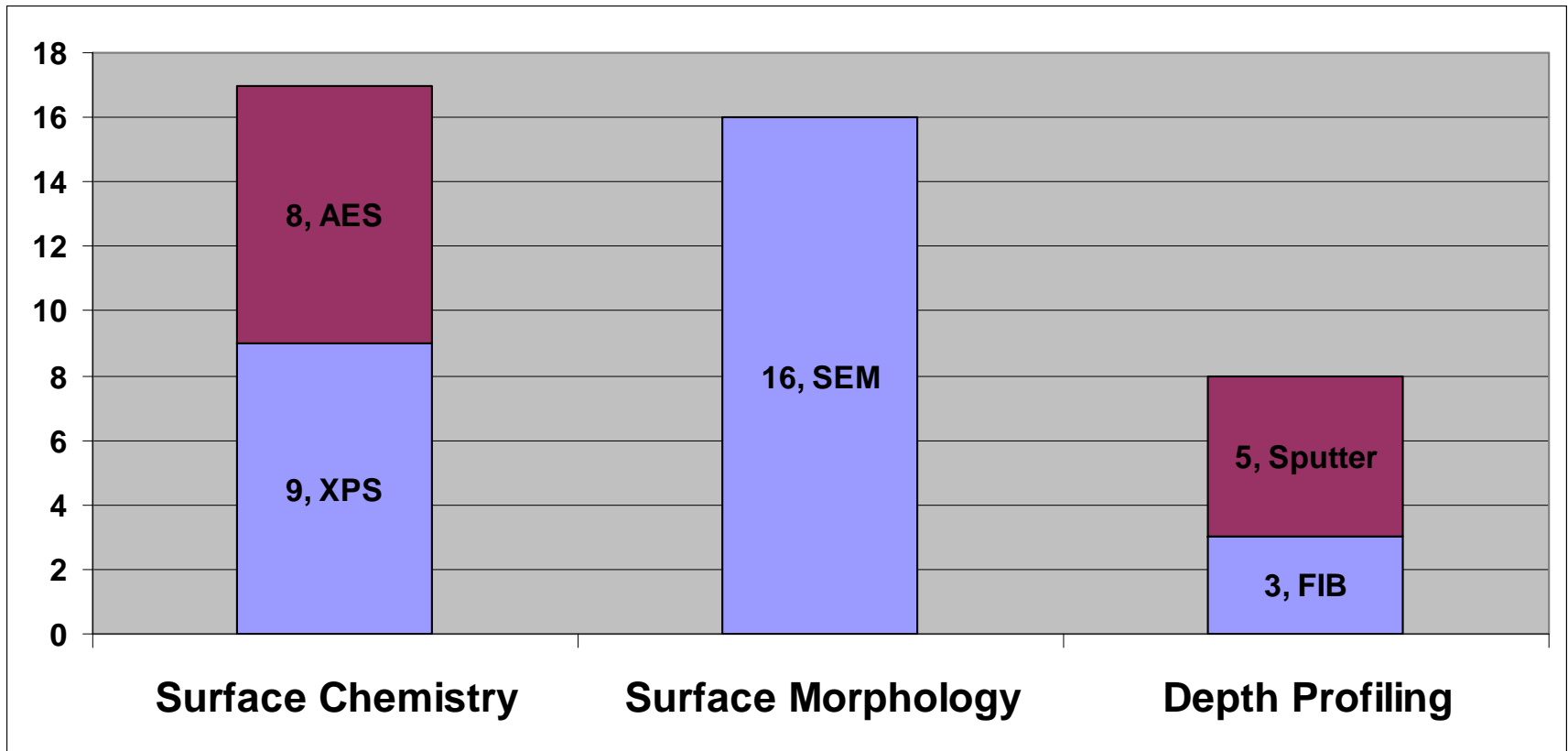
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Objective 1:

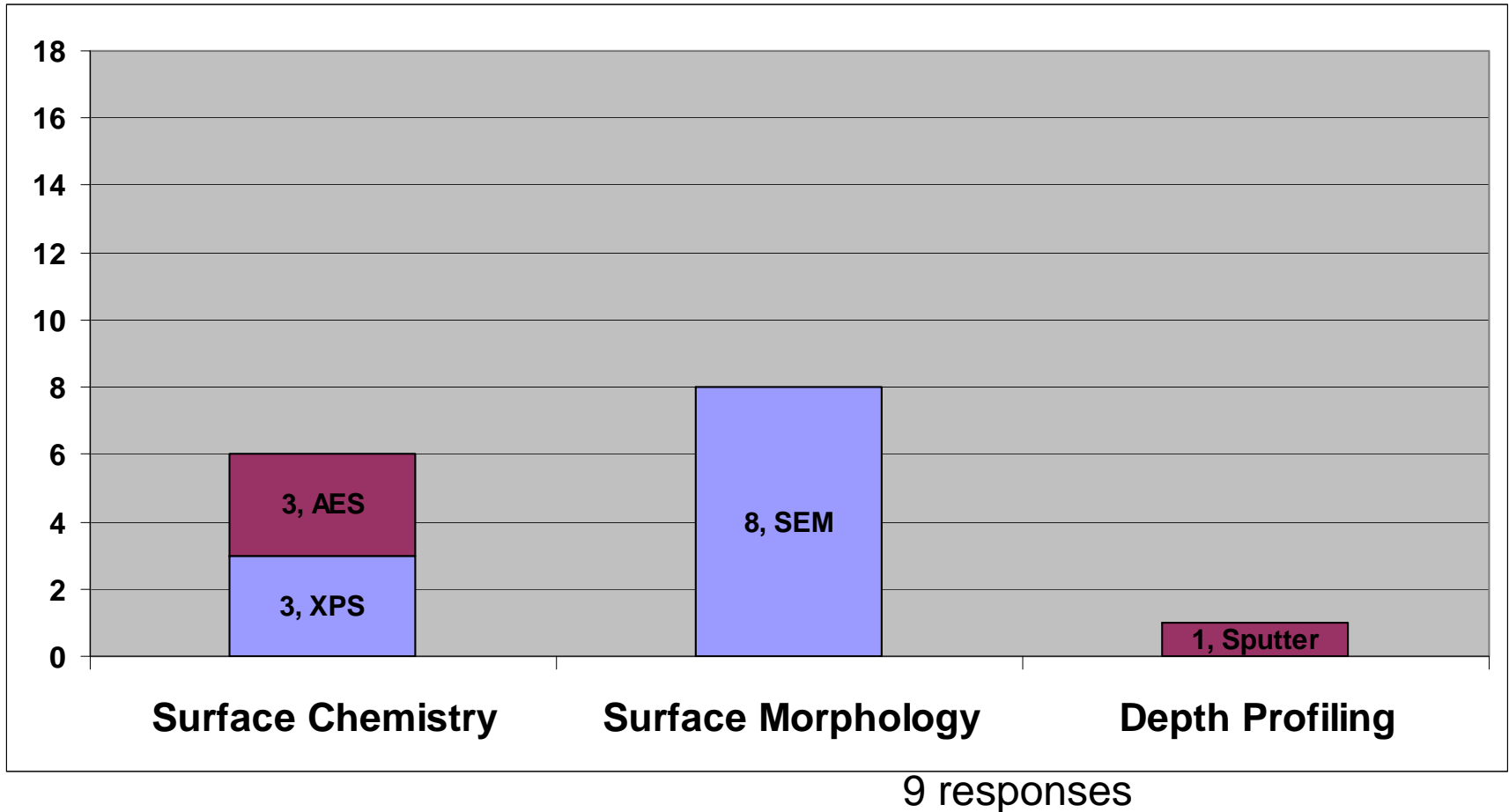
- Identify commonly used methods for surface characterization of metal implant devices
- Describe benefits and drawbacks of current surface characterization techniques
- Discuss whether surface characterization is needed in general

HW: Overview of Characterization Type and Technique Usage

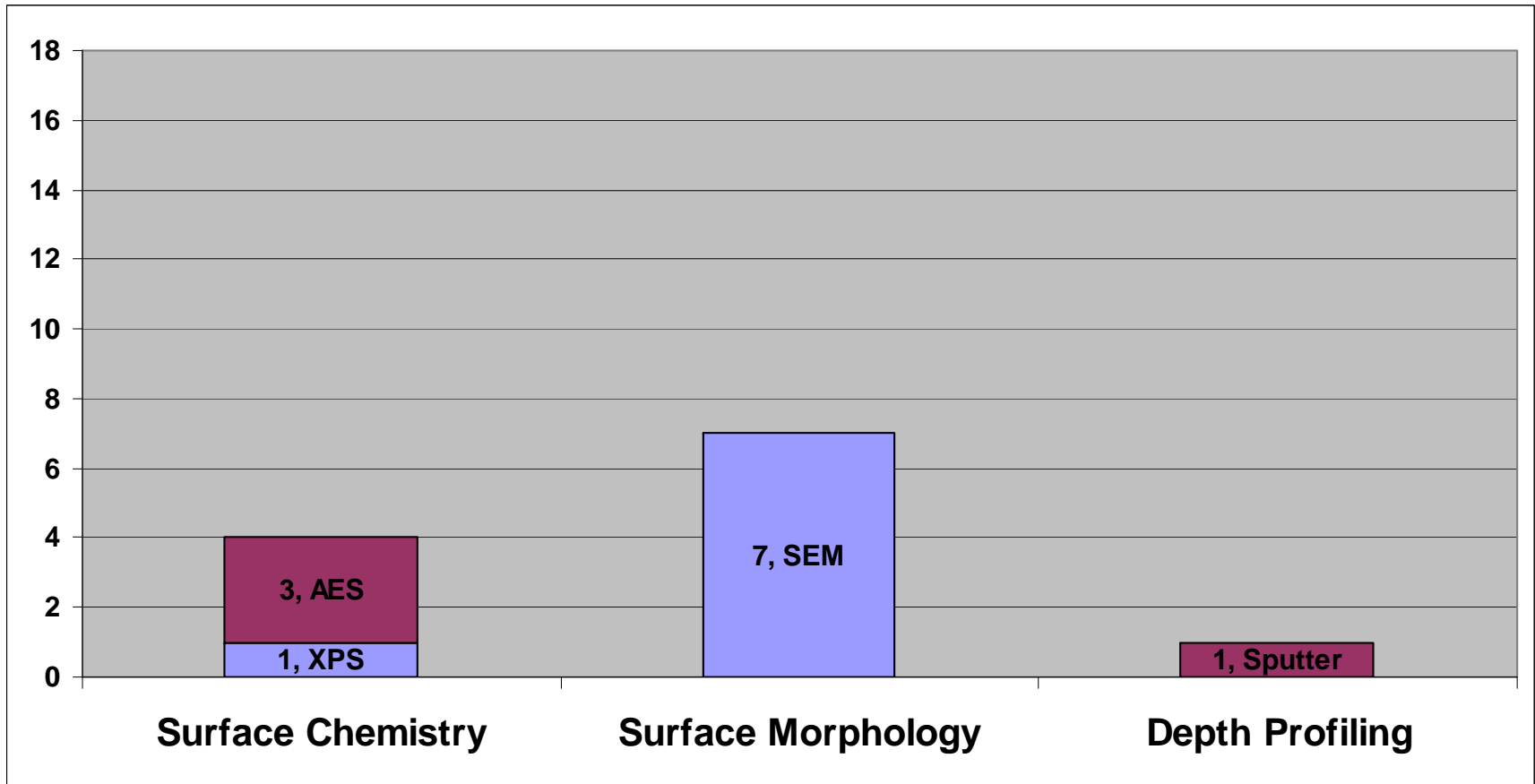


17 responses

HW: Characterization Type and Technique Usage - SS

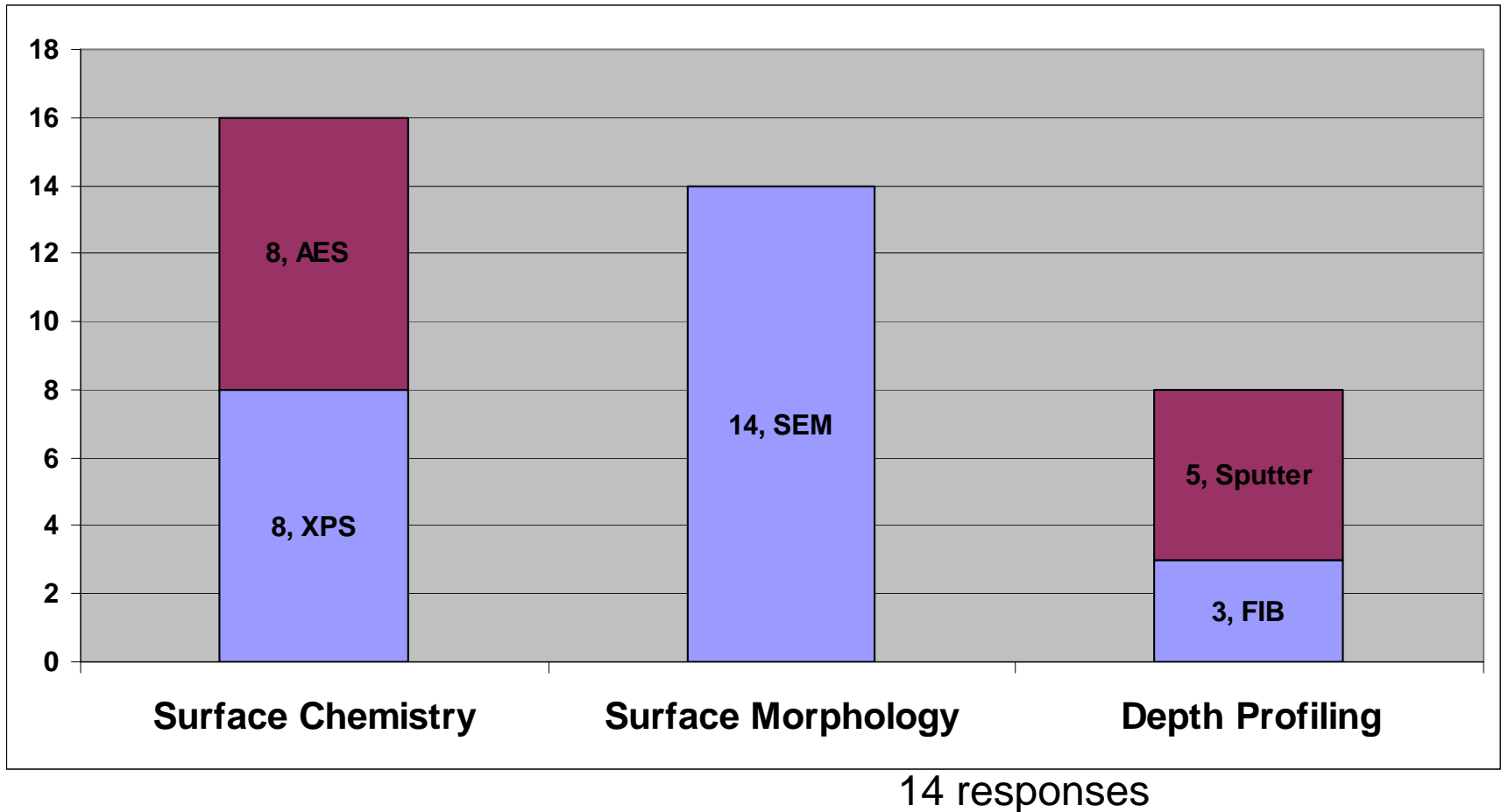


HW: Characterization Type and Technique Usage - CoCr

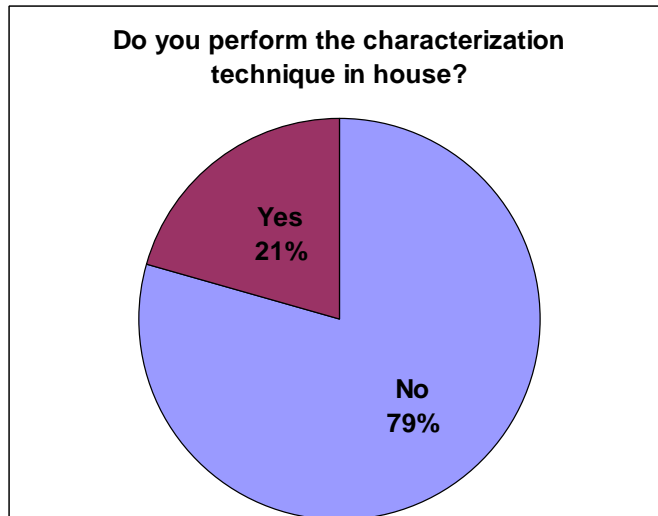
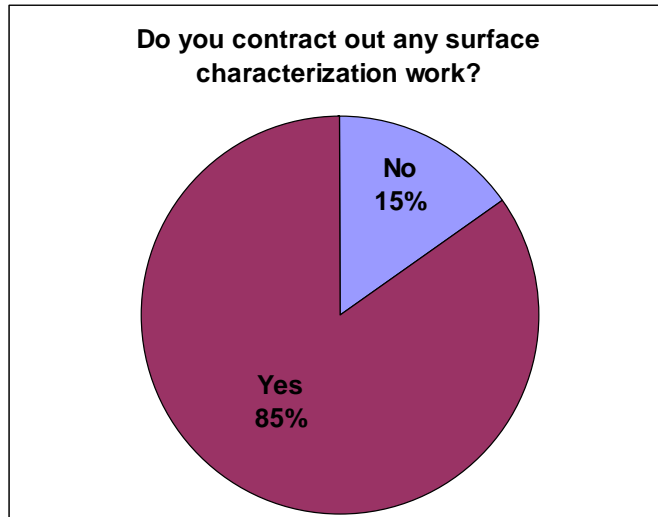


7 responses

HW: Characterization Type and Technique Usage - Nitinol



HW: In House Surface Characterization



14 Responses

- SEM most likely technique to be performed in house
- Cost of equipment and maintenance primary reason for contracting out

HW: Technique Limitations

- AES
 - Small spot size, not recommended for depth profiling
 - Coatings on finished devices can make it difficult to characterize the surface oxide
 - Surface roughness and contamination can affect oxide thickness measurements
 - Complex device geometry can mask regions
- XPS
 - Low spatial resolution
 - Coatings on finished devices can make it difficult to characterize the surface oxide
 - Samples need to be cut down
 - Some geometries hard to characterize
 - Does not inherently provide composition vs. depth
 - Poor lateral resolution (better for flat coupons)

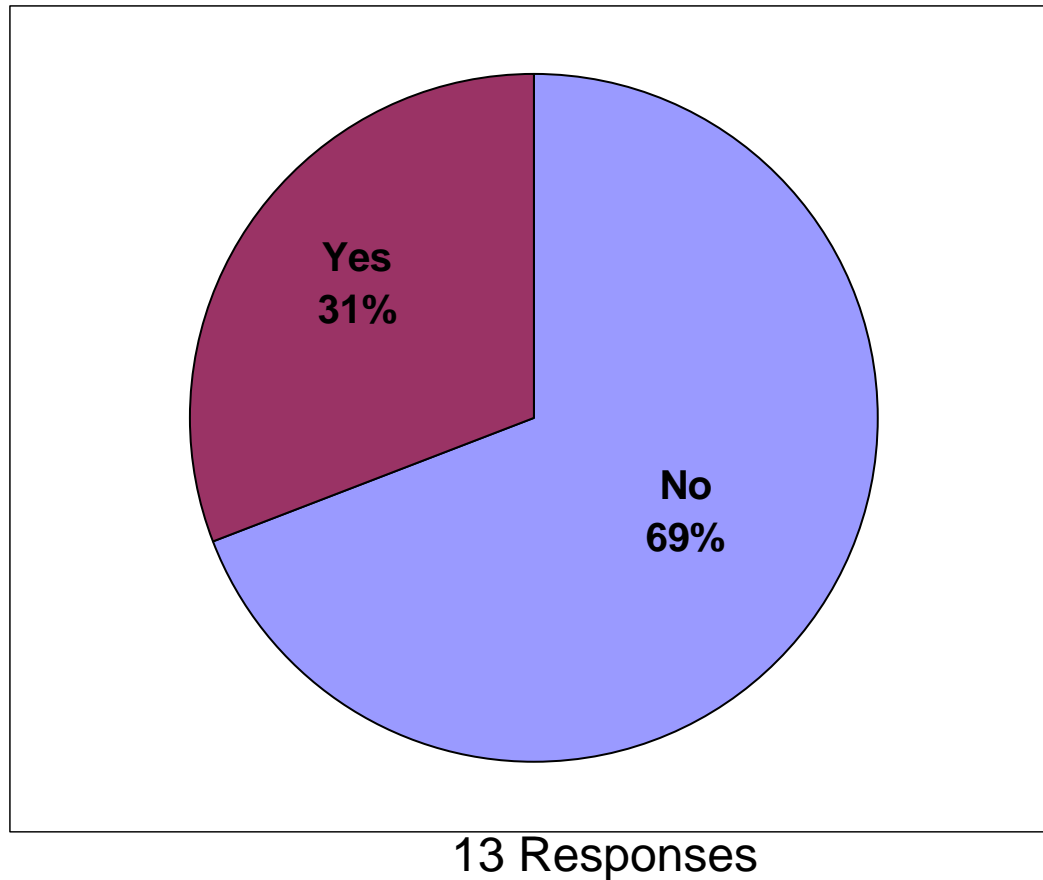
HW: Technique Limitations

- FIB/SEM
 - Does not provide information in regards to composition.
 - Does not work on thin oxides.
 - Covers a small area.
- Sputtering: AES/XPS
 - Done relative to SiO_2 which sputters at a different rate from TiO_2 .
 - Covers a small area
 - Can not be used alone to predict corrosion

HW: Reported Oxide Thicknesses

- Sputtering with AES/XPS
 - 2.8 -120 nm, 5 responses
 - Calculated by full width half maximum of oxygen peak
 - Depth calibrated to a silica standard
- FIB with SEM
 - 2.3-168 nm, 3 responses
 - Determined by visual measurement of oxide thickness
 - Dependent on SEM scale calibration and angle of sample

HW: Do you think surface characterization testing should always be performed?



Most “no” respondents recommended performing surface characterization at some point in device lifecycle (primarily R&D)

HW: When should surface characterization be performed?

- General Agreement
 - Results of performance testing are questionable (e.g. low E_b)
 - Process change that can impact the surface
 - Process/device development
- Dissension (multiple answers for and against)
 - Process/device validation
 - Routine process monitoring

Discussion Questions

- When should these techniques be used but currently are not? Conversely, when should these techniques not be used but currently are? ~10 min
- Have you observed any device characteristics/geometries that are problematic for surface characterization? ~10 min
- Is there a general role for surface characterization in process validation and/or monitoring? ~10 min

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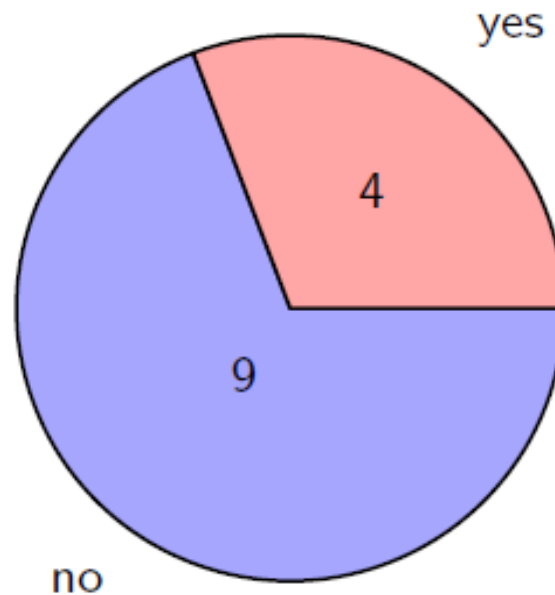
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Objective 2

Discuss the relevance and utility of surface characterization:

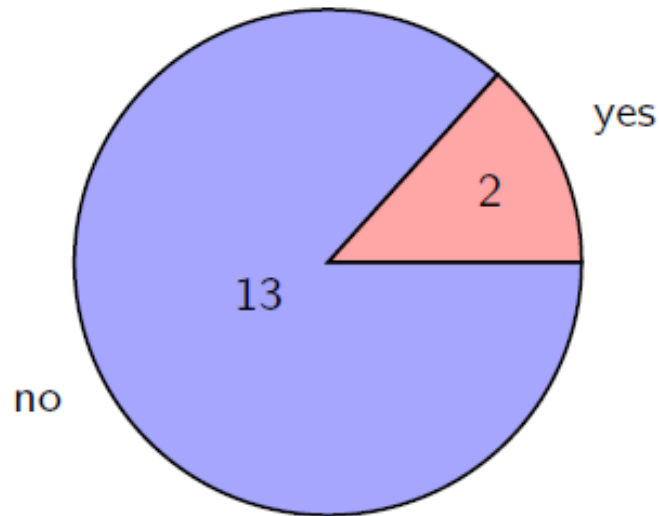
- Discuss surface structure-property relations
- Discuss potential criteria for desirable or undesirable surfaces

HW: Have you observed or identified any structure-property relations?



- Existing data for NiTi primarily represented by a few papers in the open literature that focus mainly on thermal oxides.

HW: Should there be acceptance criteria for oxide layer thickness and chemistry?



Specifics for NiTi only (oxide thickness, chemistry):

- < 15 nm, no Ni-rich phases
- < 50 nm, < 20 at.% Ni-rich regions
- < 10 nm (guideline)

Acceptance criteria are not appropriate because ...

- no established structure-property relations (in-vitro or in-vivo)
- only a small fraction of surface area analyzed
- available techniques are not adequate
- corrosion and nickel leach testing are easier and more direct

Discussion topics

1. If acceptance criteria for surface structure are currently not practical or feasible, are there examples of exclusion criteria that would be? (~15 min)
2. What are the benefits, if any, of better establishing surface structure, property relations for NiTi (or common device alloys in general)? (~15 min)